

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	H5	Oct. 7, 2022
SODA PULP INDUSTRY	Determination of residual alkali in sodium hypochlorite			

1. Abstract

Sodium hypochlorite is produced by having sodium hydroxide absorb chlorine gas. Produced sodium hypochlorite contains sodium chloride generated by the degradation of hypochlorous acid.

This report introduces an example for the determination of residual alkali (sodium hydroxide, sodium carbonate) in the sodium hypochlorite with potentiometric titration. The procedure is described as follows: Add hydrogen peroxide to sodium hypochlorite to decompose the sodium hypochlorite (reaction (1)). Run neutralization titration with hydrochloric acid standard solution (reaction (2) ~ (4)). The pH reaches at around 8 on the reaction of (2) and (3). In the case of sodium carbonate, the sodium hydrogen carbonate generated on reaction (3) is continuously titrated with hydrochloride acid, and pH reaches to around 4 at the end point on the reaction (4). The reaction from (2) to (4) and related titration curve are illustrated in figure 1.

Titration volume for sodium hydroxide can be calculated by subtracting titration volume (b mL) of 2nd end point titration from titration volume (a+b mL) of 1st end point.

The concentration of sodium carbonate can be determined by titration volume of 2nd end point. Therefore sodium hydroxide and sodium carbonate can be determined separately with successive titration. Inflection point at around pH 11.5 is expected as the end point for sodium hydroxide, but the inflection point is unclear. Therefore it is not identified as the end point on this titration.

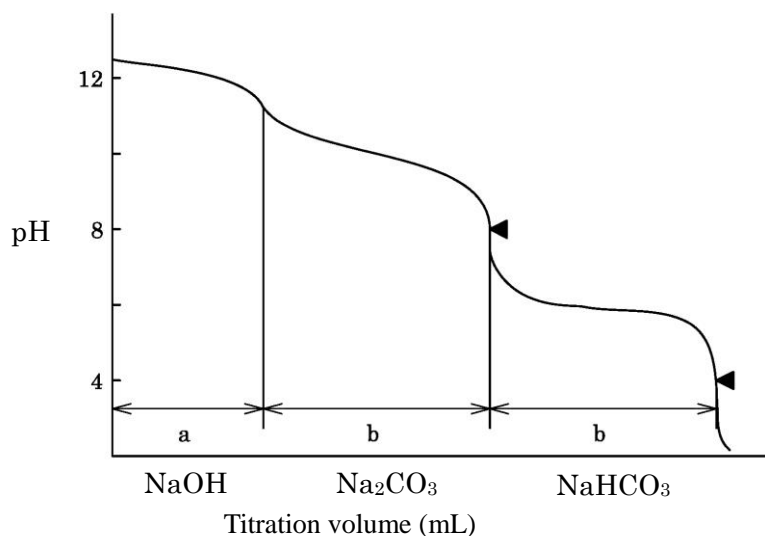
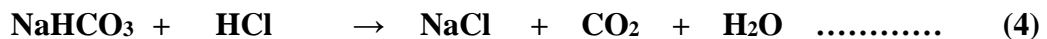
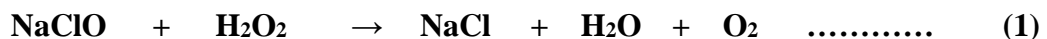


Figure 1 Neutralization titration curve for sodium hydroxide and sodium carbonate with hydrochloric acid

2. Configuration of instruments and reagents

(1) Configuration of instruments

Main unit	: Hiranuma Automatic Titrator COM series		
Electrodes	: Glass electrode	GE-101B	
	: Reference electrode	RE-201Z	

*Instead of above electrode, the following electrodes are useable.

- GR-501B (Glass reference combination electrode)···Fixed sleeve type
- GR-511B (Glass reference combination electrode)···Moveable sleeve type

<Remark> Combination with PT-301 allows to measure available chlorine in sodium hypochlorite..

(2) Reagents

Titrant	: 0.2 mol/L Hydrochloric acid standard solution
Additive solution	: 1 mL of 30 % Hydrogen peroxide

3. Measurement procedure

- (1) Dispense 2 mL of sample and weigh accurately.
- (2) Add 30 mL of DI water.
- (3) Add 1 mL of 30 % hydrogen peroxide to decompose the sodium hypochlorite.
Please note that it reacts vigorously in this procedure.
- (4) Immerse the electrodes and titrate with 0.2 mol/L hydrochloric acid standard solution.

4. Measurement conditions and results

Examples of titration conditions

(1) Measurement condition of sodium chloride and sodium carbonate

Cndt No.	1	ConstantNo.	1	Mode No.	5
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	1	Molarity	0.2 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.004	Int Time	3 sec
S-Timer	60 sec	K	40.0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula			0.05 mL
End Sens	700	(VA-VB)*K*F*M/(S*10)			
Over mL	0 mL	Decimal Places	4		
Max. Vol.	20 mL	Auto In Pram.	None		

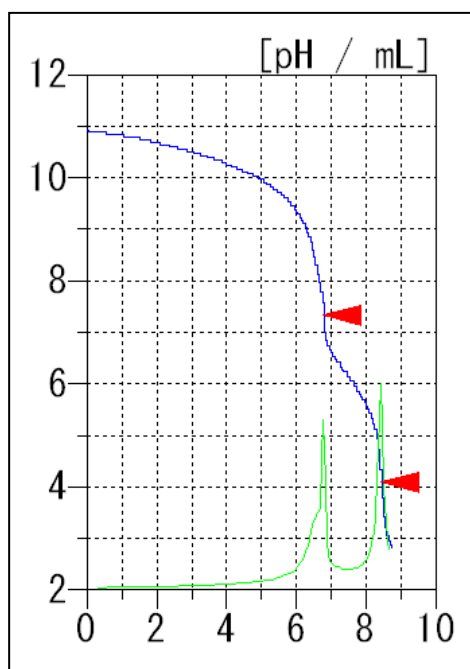
(2) Measurement condition of sodium carbonate

Cndt No.	2	Constant No.	2	Mode No.	5
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	1	Molarity	0.2 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.004	Int Time	3 sec
S-Timer	0 sec	K	105.99	Int Sens	3 mV
C.P. pH	9.0 pH	L	0	BrT Speed	2
T Timer	0 Sec	Unit	%	Pulse	40
D.P. pH	6.0 pH	Formula	(D-B)*K*F*M/(S*10)		0.05 mL
End Sens	500	Decimal Places	4		
Over mL	0.3 mL	Auto In Pram.	None		
Max. Vol.	20 mL				

<NOTE> Connect condition of (1) and (2) for the titration.

Measurement results

Number of Measurement	Size (g)	1 st End point Titrant Volume(mL)	Sodium Hydroxide (%)	2 nd End point Titrant Volume (mL)	Sodium Carbonate (%)
1	2.5456	6.770	1.6149	1.652	1.3812
2	2.5288	6.723	1.6005	1.684	1.4173
3	2.5144	6.673	1.5835	1.716	1.4525
		Avg.	1.600 %	Avg.	1.417 %
		SD	0.016 %	SD	0.036 %
		RSD	0.98 %	RSD	2.52 %



Example of titration curve

5. Note

Since the residual sodium hypochlorite could cause measurement errors, the excess hydrogen peroxide should be added to decompose sodium hypochlorite completely.

Keywords: Sodium hypochlorite, Residual alkali, Sodium hydroxide, Sodium carbonate, Neutralization titration

*Some measurement would not be possible depending on optional configuration of system.